09/913903

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PATENT COOPERATION TREATY

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

From the INTERNATIONAL BUREAU

To:

HARRISON GODDARD FOOTE
Tower House
Merrion Way
Leeds LS2 8PA
ROYAUME-UNI

Date of mailing (day/month/year) 27 août 2001 (27.08.01)	
Applicant's or agent's file reference CTV/P45135WO	**IMPORTANT NOTIFICATION**
International application No. PCT/GB00/00523	**International filing date** (day/month/year) 17 février 2000 (17.02.00)

1. The following indications appeared on record concerning:

[X] the applicant [ ] the inventor [ ] the agent [ ] the common representative

Name and Address	State of Nationality	State of Residence
NEWCASTLE UNIVERSTIY VENTURES LIMITED Sun Alliance House 35 Mosley Street, Newcastle Upon Tyne NE1 1XX United Kingdom	GB	GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

[X] the person [ ] the name [ ] the address [ ] the nationality [ ] the residence

Name and Address	State of Nationality	State of Residence
PROTENSIVE LIMITED 35 Hills Road Cambridge CB2 1NT United Kingdom	GB	GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

3. Further observations, if necessary:

4. A copy of this notification has been sent to:

[X] the receiving Office
[ ] the International Searching Authority
[ ] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland** Facsimile No.: (41-22) 740.14.35	Authorized officer R. Raissi Telephone No.: (41-22) 338.83.38

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PCT/GB00/00523

PA...NT COOPERATION TREAT...

From the INTERNATIONAL BUREAU

PCT

NOTIFICATION OF THE RECORDING OF A CHANGE

(PCT Rule 92bis.1 and Administrative Instructions, Section 422)

To:

HARRISON GODDARD FOOTE
Tower House
Merrion Way
Leeds LS2 8PA
ROYAUME-UNI

Date of mailing (day/month/year) 01 December 2000 (01.12.00)	
Applicant's or agent's file reference CTV/P45135WO	**IMPORTANT NOTIFICATION**
International application No. PCT/GB00/00523	**International filing date** (day/month/year) 17 February 2000 (17.02.00)

1. The following indications appeared on record concerning:

[X] the applicant [ ] the inventor [ ] the agent [ ] the common representative

Name and Address	State of Nationality	State of Residence
UNIVERSITY OF NEWCASTLE 6 Kensington Terrace Newcastle upon Tyne NE1 7RU United Kingdom	GB	GB
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	Facsimile No.	
	Teleprinter No.	

2. The International Bureau hereby notifies the applicant that the following change has been recorded concerning:

[ ] the person [X] the name [X] the address [ ] the nationality [ ] the residence

Name and Address	State of Nationality	State of Residence
NEWCASTLE UNIVERSTIY VENTURES LIMITED Sun Alliance House 35 Mosley Street, Newcastle Upon Tyne NE1 1XX United Kingdom	GB	GB
	Telephone No.	
	Facsimile No.	
	Teleprinter No.	

3. Further observations, if necessary:

The person in Box I has transferred the Assignment to the person in Box II.

4. A copy of this notification has been sent to:

[X] the receiving Office
[ ] the International Searching Authority
[X] the International Preliminary Examining Authority
[ ] the designated Offices concerned
[X] the elected Offices concerned
[ ] other:

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland** Facsimile No.: (41-22) 740.14.35	Authorized officer Lazar Joseph Panakal Telephone No.: (41-22) 338.83.38

Form PCT/IB/306 (March 1994)

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P ENT COOPERATION TREA

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents
United States Patent and Trademark Office
Box PCT
Washington, D.C.20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 23 October 2000 (23.10.00)	
International application No. PCT/GB00/00523	**Applicant's or agent's file reference** CTV/P45135WO
International filing date (day/month/year) 17 February 2000 (17.02.00)	**Priority date** (day/month/year) 17 February 1999 (17.02.99)
Applicant RAMSHAW, Colin et al	

1. The designated Office is hereby notified of its election made:

   [X] in the demand filed with the International Preliminary Examining Authority on:

   15 September 2000 (15.09.00)

   [ ] in a notice effecting later election filed with the International Bureau on:

2. The election [X] was [ ] was not

   made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO **34, chemin des Colombettes** **1211 Geneva 20, Switzerland** Facsimile No.: (41-22) 740.14.35	Authorized officer Catherine Massetti Telephone No.: (41-22) 338.83.38

wrong

(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication number: **0 499 362 A1**

(12) **EUROPEAN PATENT APPLICATION**

(21) Application number: **92300473.3**

(22) Date of filing: **20.01.92**

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(30) Priority: **09.02.91 GB 9102766**

(43) Date of publication of application: **19.08.92 Bulletin 92/34**

(84) Designated Contracting States: **BE CH DE DK ES FR IT LI NL SE**

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(54) **Destruction process for photocatalytically degradable organic material.**

(57) A process for the decomposition of photocatically degradable organic material includes exposing the organic material to ultraviolet light as the material is passing across the surface of a spinning disc. The surface of the disc carries anatase titanium dioxide adhering to the disc. The anatase titanium dioxide acts as a catalyst in the degradation process and preferably has a high surface area.

Organic materials such as hydrocarbons alcohols, acids, esters and others are destroyed by this environmentally acceptable process.

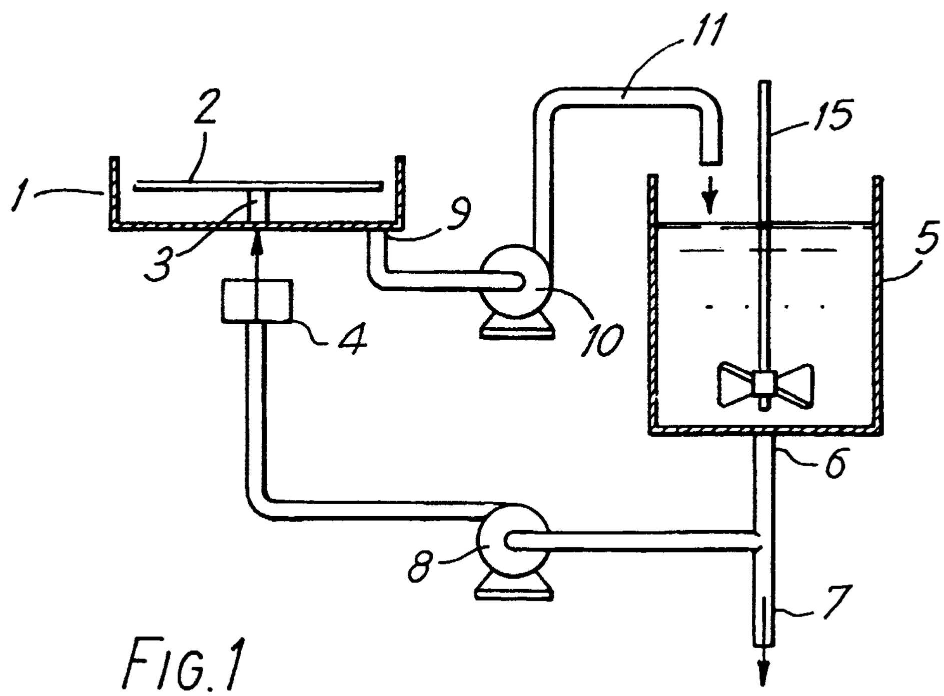

FIG. 1

EP 0 499 362 A1

This invention relates to a destruction process and particularly to a process for the decomposition of organic material by ultraviolet light.

According to the present invention a process for the decomposition of photocatalytically degradable organic material comprises exposing said organic material in fluid form to ultraviolet light and passing said organic material across the surface of a plate-like member rotating about a central axis perpendicular to the radial plane of said member thereby accelerating said organic material radially outwardly of said axis across said surface of said member which carries anatase titanium dioxide adhering to said surface.

Generally speaking this invention makes use of a so-called "spinning disc reactor". This type of reactor includes within a reaction chamber a plate-like member or an assembly of a plurality of such members which is rotated about its central axis, usually a vertical axis, but a horizontal axis, or any other orientation is not excluded, to effect transfer of a liquid material from the central axis radially across the plate or plates to agitate and disturb said liquid material. Usually the liquid will be transferred either horizontally or vertically depending on the orientation of the plate. This type of reactor has now been found to be of value in promoting the degradation of photodegradable organic materials since it is designed to maximise turbulence within a very thin liquid film. This high degree of turbulence facilitates the mass transfer of oxygen, organic entitites, reaction products and intermediates, and other reactive species across the catalyst/liquid, and liquid/gas, interfaces within the system. Most other devices incorporating an immobilised TiO_2 suffer from mass transfer limitations.

The plate-like member usually has the form of a disc and the surface which is to contact the organic material can be provided with protrusions, indentations or can be corrugated, porous or perforated. As the plate-like member is rotated liquid flows from the central axis radially outwardly across the surface of the member and is accelerated and agitated.

Usually the organic material to be treated in the process of the invention is introduced in the form of a fluid into the reactor at the centre of the plate-like member and conveniently is introduced along the axis through a support for the member which also provides the rotational drive to the plate-like member from a suitably located electric motor or other rotational drive unit, e.g. a hydraulic motor.

The plate-like member can be formed from any material which is sufficiently strong to withstand the stress generated in the material during use. Preferably the material is substantially resistant to attack by any compound with which it is brought into contact during use. Typically the plate-like member is formed of glass, ceramic or preferably a metal such as stainless steel, nickel or titanium but other materials such as wood, porous plastic and paper can be used. A borosilicate glass plate-like member has been found to be useful when the member is formed of glass.

Typically the plate-like member when in the form of a disc has a diameter of from 25 cm to 5 metres. The member can have a thickness of from 0.05 mm to 50 mm, preferably from 0.25 mm to 5 mm, especially from 0.5 mm to 2.5 mm.

If desired the plate can have a series of concentric grooves on the upper surface to be contacted with the liquid. V-shaped grooves presenting a continuously decreasing gradient to the liquid as it travels across the surface of the plate-like member increase the retention of the liquid on the surface at higher rotational speeds of the member.

Generally speaking the speed of rotation of the plate-like member is in the range 50 rpm to 10,000 rpm and preferably in the range 100 rpm to 5000 rpm. The speed of rotation affects the acceleration of the liquid across the surface of the plate-like member.

The speed of rotation and the rate of flow of liquid onto the surface of the plate-like member are such that a thin film of the liquid is formed on the rotating surface of the member and this thin film is subjected during rotation to a high degree of turbulence as it is thrown radially outwardly of the member.

Normally the plate-like member is mounted with its surface either vertical or horizontal and it is the upper surface across which the liquid is caused to flow during exposure to ultraviolet light.

The surface of the member in contact with the liquid carries a photoactive catalyst which promotes the degradation of the organic material. In the process of the present invention the catalyst is anatase titanium dioxide, typically that produced by the hydrolysis of a soluble titanium compound such as titanyl sulphate or titanium tetrachloride and which after precipitation is calcined to produce the anatase titanium dioxide. Preferably the calcination conditions are chosen so that the time and/or temperature is somewhat less than that which would be required to produce optimum pigmentary anatase titanium dioxide. The catalyst preferably has a high surface area of from 20 to 200 m^2/gm. Typically a hydrated precipitate of titanium dioxide is calcined at a temperature of from 100°C to 1000°C for 10 minutes to 1000 minutes. Usually the anatase titanium dioxide has a particle size of from 0.001 micron to 1.0 micron.

If desired the anatase titanium dioxide can be produced by the oxidation of a titanium halide such as titanium tetrachloride under conditions such that the product has the desired high surface area.

The plate-like member carries the active catalyst at least on the surface to be in direct contact with the liquid

to be treated and, as a result of the method of preparation, usually both radial surfaces of the member are coated with the chosen catalyst. One suitable procedure employed to coat the member with the catalyst is to immerse the member in an aqueous dispersion of the anatase titanium dioxide for a period of say 3 to 10 minutes and then dry the treated plate member in an oven for a period of say 30 to 75 minutes at a temperature of 70°C to 100°C. This treatment procedure is repeated until a desired effective amount of the catalyst has been applied to the surface of the member. Using an aqueous dispersion containing from 5 to 15 gram per litre TiO_2 a total of from 7 to 15 immersion/drying cycles produces an "active" member.

Other procedures can include immersion in solutions of organic titanium compounds, with precipitation of TiO_2 by sol/gel techniques, and pyrolysis of titanium compounds directly onto the surface of the member. Additionally other support materials can be coated with TiO_2 and then attached to the surface of the member.

The organic material to be treated in the process of the invention is in the form of a fluid during treatment. Where the organic material to be degraded is a liquid itself then it can be treated directly. However the organic can be dissolved or dispersed in water or in any other suitable medium prior to treatment. Aqueous solutions are preferred since the presence of water acts as a source for hydroxyl radicals and facilitates the transport of oxygen across the liquid/gas and solid/liquid interface. Typically the aqueous solution of the organic can have any pH value but preferably is acidic having a pH less than 7 and more preferably less than 4.

Activation of the anatase titanium dioxide catalyst is ensured by exposing the catalyst to the effect of ultraviolet light. The liquid to be treated is exposed to the light as it is in contact with the surface of the plate-like member and whilst ultraviolet light of any wavelength can be used it has been found that light emitted by so-called low pressure lamps is more effective in promoting degradation of the organic material. Typically UV light of up to 400 nanometers can be used but the most preferred light is that having a wavelength of from 240 to 280 nm.

The process can be operated batchwise or continuously. In batch operation liquid to be treated is held in a holding tank and recycled across the surface of the rotating plate member until all necessary degradation has been completed. Alternatively continuous operation can be effected if the required degradation is obtained by a single pass across the surface of the plate member or by a succession of passes across a number of different plate members. Usually suitable analytical means will be employed to test the extent of degradation prior to discharge of water to the environment.

Any organic compound which is capable of photodegradation can be treated by the method of the invention. Depending on the exact nature of the organic material various by-products can be obtained. For those organic compounds composed solely of carbon hydrogen and oxygen the process produces water and carbon dioxide as the degradation products. For organic materials containing halogen additionally dilute mineral acid is a degradation product. The process, in any event, produces relatively easily handleable chemicals from often complex organic compounds.

Usually the process of the invention is carried out at room temperature with the rotating plate mounted in a suitable confining reactor equipped with a suitable source of ultraviolet light.

Typical organic compounds which can be treated in accordance with the invention are aliphatic or aromatic hydrocarbons, alcohols, acids, esters, ketones, amines and halogen substituted compounds. Pesticides are other environmentally hazardous organic products eminently suitable for treatment by the process of the invention.

The invention is illustrated in the following Examples in which apparatus as shown in the accompanying drawing was used.

In the drawing:

Figure 1 is a diagrammatic representation of the overall layout,

Figure 2 is one form of reactor, and

Figure 3 is an alternative form of reactor.

As shown the apparatus includes a reactor chamber 1 having mounted horizontally therein a rotatable disc 2 on a hollow shaft 3 coupled to a motor 4. A storage tank 5 has an outlet 6 in the base of the tank 5 through which the contents of the tank can be drained through pipe 7. The outlet 6 is also coupled to a pump 8 to feed the contents of the tank 5 through the hollow shaft 3 to the upper surface of the disc 2. The base of the reactor chamber 1 has an outlet 9 to a pump 10 and a return pipe 11 to the tank 5.

Figure 2 illustrates one form of reactor chamber 1 in which there is horizontally mounted lamps 12 to produce ultraviolet light. The lamp 12 extends across a diameter of the disc 2.

In Figure 3 an alternative arrangement of reactor chamber 1 is shown in which the lamp 12 is mounted vertically above but axially in line with the axis of the disc. A reflector 13 is positioned to direct the light onto the disc 2.

The reactor chamber 1 is equipped with an axial deflector plate 14 to deflect flow of liquid from the hollow shaft 3 onto the upper surface of the disc 2. The tank 5 is equipped with a stirrer 15.

As used the rotatable disc 2 was formed from borosilicate glass and had a diameter of 38 cm. The speed of rotation of the disc in the following experiments was 350 rpm and a liquid flow rate across the upper surface of the disc 2 was maintained at 180 litres per hour. The temperature within the reactor chamber 1 was maintained at about 25°C.

The rotatable disc 2 carried a coating of anatase titanium dioxide the particular form of which is described in the following examples. The disc was coated by preparing an aqueous slurry of the titanium dioxide containing 10 gpl by milling the anatase titanium dioxide with water in the required amount and the disc 2 was then immersed in the slurry for a period of 5 minutes. The disc was removed from the slurry and dried in an oven at 90°C for one hour. This particular coating and drying procedure was repeated for a total of 10 cycles. The disc was then washed thoroughly after the last drying stage to remove any loose titanium dioxide particles from the surface.

Example 1

A 38 cm disc of borosilicate glass was coated with a thin film of TiO_2 as described previously.

The disc was attached to the shaft 3 and run at 350 rpm. An aqueous solution containing 100 micromoles per litre of 4-chlorophenol was pumped over the disc at the rate of 180 l/hr whilst the disc was illuminated with UV light as shown in Figure 2 from two 15 watt low-pressure lamps at different intensities. Solution pH was maintained at pH 3.1 with 2% H_2SO_4.

Rates of reaction calculated from experimental data were as follows:-

	UV intensity Wm^{-2}	Rate of Reaction (K_R) Micromoles/min/litre
Experiment A	14.0	0.563
Experiment B	27.9	0.652

K_R is defined by reference to the Langmuir-Hinshelwood Kinetics.

Example 2

Experimental conditions were similar to Example 1 except that the low pressure lamp was substituted by a 400 watt medium pressure lamp as in Figure 3.

Results were as follows:

	UV intensity Wm^{-2}	Rate of Reaction (K_R) Micromoles/min/litre
Experiment A	27.9	0.228
Experiment B	49.0	0.245
Experiment C	98.2	0.381
Experiment D	246.0	0.418

The above results demonstrate that the use of a low pressure lamp increases the speed of destruction of 4-chloro-phenol, compared with a medium pressure, higher output, lamp. This increase could not be accounted for in terms of a photochemical reaction.

Example 3

Examples 1 and 2 were repeated with an initial concentration of 100 micromoles/litre of salicylic ac4id.

	UV Intensity Wm-2	Lamp	Rate of Reaction (K_R) Micromoles/min/litre
Experiment A	27.9	Low Pressure	0.419
Experiment B	246.0	Medium Pressure	0.185

The rate of degradation of the salicylic acid is slower than for 4-chlorophenol, but, once again, the low pressure lamp is more effective than the medium pressure lamp.

EXAMPLE 4

Experimental conditions were similar to Experiment 1 using two 15W low pressure lamps and an initial concentration of 100 micromoles per litre of 4-chlorophenol. Solution pH was controlled by addition of acid or alkali as required.

	Solution pH	Organic	Rate of Reaction Micromoles/min/litre
Experiment A	3	Chlorophenol	0.418
Experiment B	5	Chlorophenol	0.257
Experiment C	11	Chlorophenol	0.121

Claims

1. A process for the decomposition of photocatalytically degradable organic material comprising exposing said organic material in fluid form to ultraviolet light and passing said organic material across the surface of a plate-like member rotating about a central axis perpendicular to the radial plane of said member thereby accelerating said organic material radially outwardly of said axis across said surface of said member which carries anatase titanium dioxide adhering to said surface.

2. A process according to claim 1 in which the anatase titanium dioxide has a surface area of from 20 to 200 m^2/gm.

3. A process according to claim 1 or 2 in which the anatase titanium dioxide has a particle size of from 0.001 to 1.0 micron.

4. A process according to claim 1, 2 or 3 in which the ultraviolet light is that emitted by a low pressure lamp.

5. A process according to any one of claims 1 to 4 in which the ultraviolet light has a wavelength of up to 400 nanometers.

6. A process according to any one of claims 1 to 6 in which the plate-like member has the form of a disc having a diameter of from 25 cm to 5 metres.

7. A process according to claim 6 in which the plate-like member has a thickness of from 0.05 mm to 50 mm.

8. A process according to any one of claims 1 to 7 in which the plate-like member is rotated at a speed of from 50 rpm to 10,000 rpm.

9. A process according to any one of the preceding claims in which the said organic material is a dispersion or a solution in water.

10. A process according to claim 9 in which the water dispersion or solution is acidic.

11. A process according to claim 10 in which the water dispersion or solution has a pH of less than 4.

12. A process according to any one of the preceding claim in which the said organic material is a hydrocarbon, an alcohol, an acid, an ester, a ketone, an amine or a halogen substituted compound.

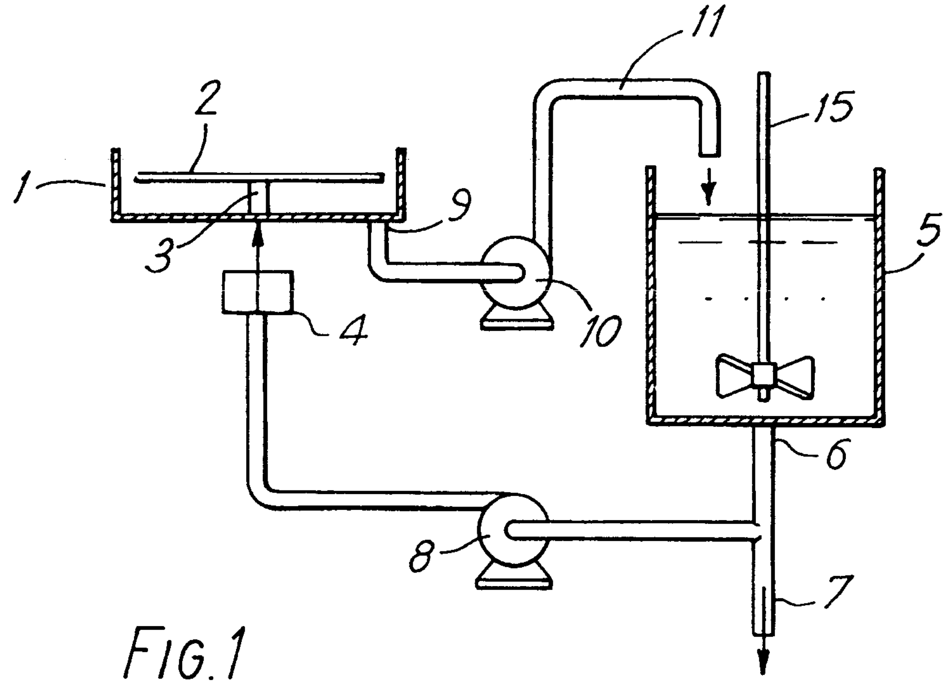

FIG.1

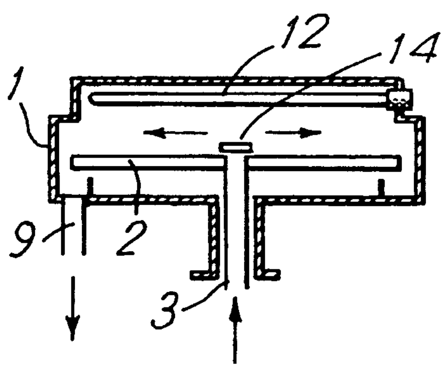

FIG.2

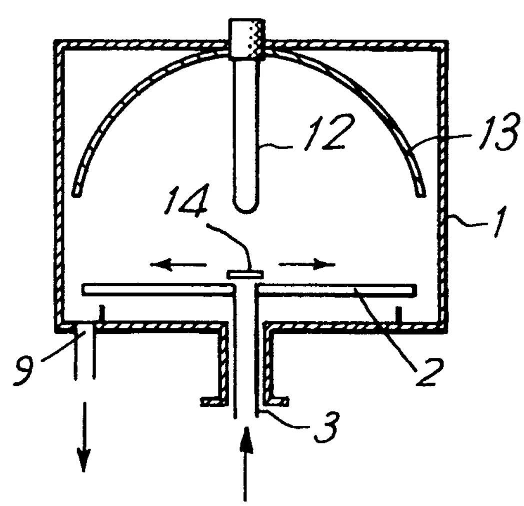

FIG.3

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 92 30 0473

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
Y	FR-A-2 450 612 (F.BOHNENSIEKER) * page 1, line 4 - page 2, line 5 * * page 3, line 23 - line 35 * * page 6, line 13 - line 35; claims 1,2,10 * ---	1,5-12	A62D3/00 B01J19/12 B01J19/18
Y	EP-A-0 306 301 (R.B.HENDERSON) * the whole document * ---	1,5-12	
A	AUSTRALIAN JOURNAL OF CHEMISTRY vol. 40, no. 4, 1987, EAST MELBOURNE AU pages 667 - 675; R.W.MATTHEWS: 'Carbon Dioxide Formation from Organic Solutes in Aqueous Suspensions of Ultraviolet-Irradiated TiO2. Effect of Solute Concentrations' * page 668, line 1 - page 670; figure 3 * * page 672, line 9 - line 21 * ---	1-5,9-12	
A	WO-A-8 900 985 (WISCONSIN ALUMNI RESEARCH FOUNDATION) * page 4, line 8 - page 10, line 2 * ---	1,5,9-12	TECHNICAL FIELDS SEARCHED (Int. Cl.5)
A	GB-A-328 410 (E.BUHTZ) * page 1, line 66 - page 2, line 19; claims * ---	1,5-9,12	A62D B01J
A	ENVIRONMENTAL SCIENCE AND TECHNOLOGY. vol. 19, no. 6, 1985, EASTON, PA US pages 480 - 484; D.F.OLLIS: 'Contaminant Degradation in Water' -----		

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	19 MAY 1992	FLETCHER A.S.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB00/00523

I. Basis of the report

1. With regard to the **elements** of the international application *(Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)):*

Description, pages:

1,3-17	as originally filed			
2	as received on	16/03/2001	with letter of	16/03/2001

Claims, No.:

1-26	as received on	16/03/2001	with letter of	16/03/2001

Drawings, sheets:

1/3-3/3	as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)

6. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	
	No:	Claims	1 - 7, 14-16, 19, 20, 24, 26
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1 - 26
Industrial applicability (IA)	Yes:	Claims	1 - 26
	No:	Claims	

2. Citations and explanations
see separate sheet

INTERNATIONAL PRELIMINARY EXAMINATION REPORT - SEPARATE SHEET International application No. PCT/GB00/00523

Section V:

1. Reference is made to the following document which is considered as closest prior art document:

   D1: US-A-5 624 999

2. Document D1 discloses a spinning wheel reactor comprising a rotor and a stator in close proximity to the surface of the rotor. The width of the gap between the stator and the rotor determines the film thickness (column 14, lines 53-5) and the reactant flows across the surface as a thin film and is then thrown from the periphery thereof. The stator inevitably imparts a shearing force to the reactant on the surface of the rotor when it is rotated (see D1, Figs. 3, 4 in connection with the corresponding description in columns 25/26 and column 14, lines 20-36).

3. The subject-matter of claims 1-7, 14-16, 19, 20, 24 and 26 appears to lack novelty in view of D1.

4. The features of the remaining dependent claims are related to features that are common in the art and therefore cannot support an inventive step. Therefore it is not apparent how the features of the dependent claims or the description could support an inventive step.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 00/00523

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5624999 A	29-04-1997	BR 9205732 A	23-08-1994
		DE 69228181 D	25-02-1999
		DE 69228181 T	15-07-1999
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US 3831907 A	27-08-1974	GB 1356921 A	19-06-1974
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		GB 1356922 A	19-06-1974
US 4511414 A	16-04-1985	JP 1520888 C	29-09-1989
		JP 60033284 A	20-02-1985
		JP 64000359 B	06-01-1989
		CA 1214643 A	02-12-1986
		DE 3462784 D	30-04-1987
		EP 0134107 A	13-03-1985
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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 00/00523

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B01J19/18 B01J19/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B01J B01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 624 999 A (LOMBARDI ALESSANDRO, BARINI GERALDO, D'ANTONIO CARMINE, GUSI STEFANO) 29 April 1997 (1997-04-29) column 14, line 20 -column 18, line 25 column 25, line 62 -column 27, line 2 figures 3-6 ---	1-7, 14-16, 19,20, 24,26
X	US 3 831 907 A (CLAES FRANS HENRI) 27 August 1974 (1974-08-27) column 4, line 36 -column 5, line 10 column 6, line 29 - line 36 column 7, line 7 - line 19 column 7, line 49 -column 8, line 27 figures 1,4 --- -/--	1,8,9, 14,15, 19-26

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
3 August 2000	11/08/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Vlassis, M

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 00/00523

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 511 414 A (MATSUI FUMIO,AONO SHIN,SAKAI HIROSHI,HATTORI KATSUHIDE,KAKINO SHIGERU) 16 April 1985 (1985-04-16) column 4, line 26 - line 61 claim 1; figure 3 ---	1-3,24
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(3)

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

HARRISON GODDARD FOOTE
Tower House
Merrion Way
Leeds LS2 8PA
GRANDE BRETAGNE

10.MAY2001*054128

PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing (day/month/year)	08.05.2001
Applicant's or agent's file reference GTY/P45135WO	[illegible]
Applicant NEWCASTLE UNIVERSITY VENTURES LIMITED et al.	

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.

2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.

3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. **REMINDER**

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   Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

   For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/	Authorized officer
European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Gregoire, J-P Tel.+49 89 2399-8041

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PATENT COOPERATION TREATY

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

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Applicant's or agent's file reference	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)
CTV/P45135WO		
International application No. PCT/GB00/00523	International filing date *(day/month/year)* 17/02/2000	Priority date *(day/month/year)* 17/02/1999
International Patent Classification (IPC) or national classification and IPC B01J19/18		
Applicant NEWCASTLE UNIVERSITY VENTURES LIMITED et al.	10.MAY2001*054129	.11

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

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3. This report contains indications relating to the following items:

   I ☒ Basis of the report
   II ☐ Priority
   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   IV ☐ Lack of unity of invention
   V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   VI ☐ Certain documents cited
   VII ☐ Certain defects in the international application
   VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
15/09/2000	08.05.2001
Name and mailing address of the international preliminary examining authority: European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Buesing, G Telephone No. +49 89 2399 8356

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(60) Parent Application or Grant
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Published

(54) Title: ROTATING SURFACE OF REVOLUTION REACTOR WITH SHEARING MECHANISMS

(54) Titre: SURFACE TOURNANTE DE REACTEUR ROTATIF COMPORTANT DES DISPOSITIFS DE CISAILLEMENT

(57) Abstract

A reactor including a rotatable disc (3) having a surface (5) onto which reactant (15) is supplied by way of a feed (4). The disc (3) is rotated at high speed, and the reactant (15) spills over the surface (5) so as to form a film (17). A shear member (18, 20) is mounted close to the surface (5) so as to contact the film (17) during operation of the reactor, thereby applying a shearing force to the reactant (15) so as to aid mixing.

(57) Abrégé

L'invention concerne un réacteur comportant un disque tournant (3) qui présente une surface (5) sur laquelle un réactif est appliqué au moyen d'un dispositif d'apport (4). On fait tourner le disque (3) à grande vitesse, et le réactif (15) se répand sur la surface (5) de manière à former un film (17). Un élément de cisaillement (18, 20) monté à proximité de la surface (5) de façon à entrer en contact avec le film (17) au cours de l'exploitation du réacteur, permet d'appliquer une force de cisaillement au réactif (15) pour faciliter le mélange.

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(71) Applicant (for all designated States except US): UNIVERSITY OF NEWCASTLE [GB/GB]; 6 Kensington Terrace, Newcastle upon Tyne NE1 7RU (GB).

(72) Inventors; and
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(74) Agent: HARRISON GODDARD FOOTE; Tower House, Merrion Way, Leeds LS2 8PA (GB).

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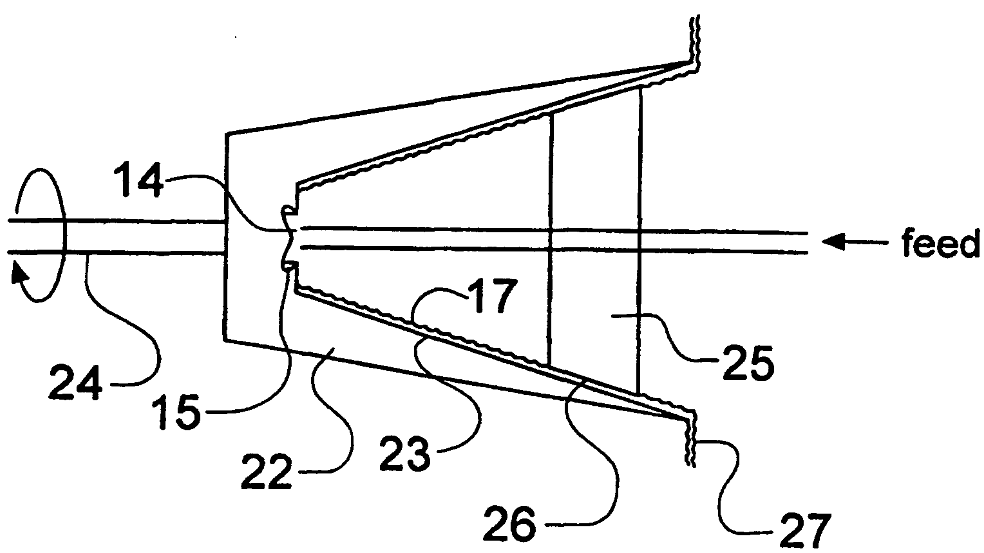
(54) Title: ROTATING SURFACE OF REVOLUTION REACTOR WITH SHEARING MECHANISMS



WO 00/48730 A3

(57) Abstract: A reactor including a rotatable disc (3) having a surface (5) onto which reactant (15) is supplied by way of a feed (4). The disc (3) is rotated at high speed, and the reactant (15) spills over the surface (5) so as to form a film (17). A shear member (18, 20) is mounted close to the surface (5) so as to contact the film (17) during operation of the reactor, thereby applying a shearing force to the reactant (15) so as to aid mixing.

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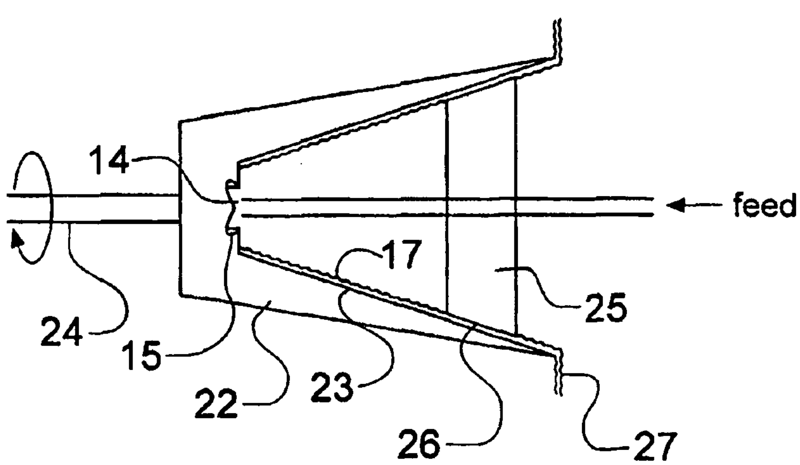

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Description

ROTATING SURFACE OF REVOLUTION REACTOR WITH SHEARING MECHANISMS

The present invention relates to a rotating surface of revolution reactor provided with various shearing mechanisms.

The invention makes use of rotating surfaces of revolution technology (hereinafter RSORT) (commonly known as spinning disc technology).

The spinning disc concept is an attempt to apply process intensification methods within the fields of heat and mass transfer. The technology operates by the use of high gravity fields created by rotation of a disc surface causing fluid introduced to the dire surface at its axis to flow radially outward under the influence of centrifugal acceleration in the form of thin often wavy films. Such thin films have been shown to significantly improve the heat and mass transfer rates and mixing. The technology was developed for typical heat and mass transfer operations such as heat exchanging, heating, cooling and mixing, blending and the like, for example as disclosed in R J J Jachuck and C Ramshaw, "Process Intensification: Heat transfer characteristics of tailored rotating surfaces", Heat Recovery Systems & CHP, Vol. 14, No 5, p475-491, 1994.

More recently the technology has been adapted for use as a reacting surface for systems which are heat and mass transfer limited, for example for the reaction of substrates which are highly viscous during at least a stage of the reaction and cause problems in achieving good mixing and product yields.

Boodhoo, Jachuck & Ramshaw disclose in "Process Intensification: Spinning Disc Polymeriser for the Manufacture of Polystyrene" the use of a spinning disc apparatus in which monomer and initiator is reacted by conventional means to provide a pre-polymer which is then passed across the surface of a spinning disc at elevated temperature providing a conversion product in the form of polymerised styrene.

EP 0 499 363 (Tioxide Group Services Limited) discloses another use for spinning disc technology in photo catalytic degradation of organic materials such a hydrocarbons. A solution of salicylic acid and titanium dioxide catalyst was passed across the surface of a rotating disc and irradiated with ultra violet light.

These publications therefore disclose the use of spinning disc technology for heating and mass transfer in inert and reactive systems.

GB 9903474.6 (University of Newcastle), from which the present application claims priority and the disclosure of which is hereby incorporated into the present application by reference, describes the use of RSORT in the conversion of a fluid phase substrate by dynamic heterogeneous contact with an agent. In this application, it is described how it has surprisingly been found that spinning disc technology may be further adapted to apply process intensification methods not only within the fields of heat and mass transfer but also within the field of heterogeneous contacting. Furthermore, it is described how it has surprisingly been found that the quality of the product obtained is of higher quality than that obtained by conventional processing having, for example, a higher purity or, in polymers, a narrower molecular distribution.

In addition to this, spinning disc technology can be used to obtain products not readily obtainable by other technology.

According to the present invention, there is provided a reactor apparatus including a support element adapted to be rotatable about an axis, the support element having a surface and feed means associated therewith for supplying at least one reactant to the surface, characterised in that there is further provided a shear member which is disposed in close proximity to the surface so as, in use, to impart a shearing force to the reactant on the surface when the support element is rotated.

It is to be understood that the term "reactant" is not limited to substances which are intended to undergo chemical reaction on the first surface of the support element, but also includes substances which are intended to undergo physical or other processes such as mixing or heating. Similarly, the term "product" is intended to denote the substance or substances which are collected from the first surface of the support element, whether these have undergone chemical or physical processing or both. In addition, although it is envisaged that most reactants and products will be in the liquid phase, the apparatus can be used with any suitable fluid phase reactants and products, including combinations of liquid, solid and gaseous reactants and products. For example, solid phase substances in substantially free-flowing particulate form can have macroscopic fluid flow properties.

An RSORT apparatus (commonly known as a spinning disc reactor) generally includes within a conversion chamber a rotating surface or an assembly of a plurality of these which is rotated about an axis to effect transfer of one or more reactants from the axis preferably radially across the rotating surface.

An RSORT apparatus as hereinbefore defined comprising a rotating surface as hereinbefore defined has a number of advantageous constructional features according to the present invention.

When the support element of the present invention is rotated at an appropriate speed, reactant applied to the surface will tend to spread out on the surface so as to form a film. As the support element continues to rotate, product will be thrown from a periphery of the surface and reactant will migrate from the point of application towards the periphery. Various chemical and physical processes may be undergone by the reactant as it migrates across the surface, as described for example in GB 9903474.6. Reactant is generally applied by the feed means at least to a central portion of the surface, and optionally to other parts thereof.

The shear member may take a number of different forms. The important consideration is that the shear member must be close enough to the rotating surface so as to apply a shearing force to the film of reactant or product located thereon. In one embodiment, the rotating surface is generally planar, and preferably disc shaped (although other shapes, such as polygons or other regular or irregular shapes, are also usable). Here, the shear member may comprise at least one generally elongate member, which may be stationary with respect to the rotating surface or which may also rotate with or against the rotating surface or otherwise move, provided that there is relative movement between the rotating surface and the elongate member during operation of the reactor. Alternatively, the shear member may comprise a generally planar surface of any appropriate size or shape, which again may be fixed relative to the rotating surface or which may also rotate with or against the rotating surface or otherwise move, provided that there is relative movement between the rotating surface and the planar surface during operation of the reactor. In yet a further embodiment, the shear member may comprise a dome or canopy of any appropriate size or shape, the dome or canopy having a peripheral basal surface which contacts the film of reactant or product on the rotating surface during operation of the reactor. The dome or canopy may be fixed relative to the rotating surface or may also rotate with or against the rotating surface or otherwise move, provided that there is relative

movement between the rotating surface and the basal surface during operation. Instead of a dome or canopy, a cylindrical or tubular member (generally of circular cross-section although polygonal and other regular and irregular cross-sections may also be of use) having a peripheral basal surface as above may be provided. The cylindrical or tubular member may be of constant cross-section along its length, or may converge, diverge or otherwise change its cross-section along its length, and may be stationary with respect to the rotating surface or may rotate with or against the rotating surface or otherwise move, as described above.

In embodiments where the shear member is rotatable, the shear member may rotate about the same axis of rotation as the rotating surface, or may rotate about an offset but generally parallel axis so as to have an orbital motion with respect to the rotating surface. The shear member may rotate in an opposite direction from the rotating surface, or may rotate in the same direction but at a different rotational speed. Where the shear member rotates about an offset axis, then it may be rotated in the same direction and at the same speed as the rotating surface while still providing the required shearing forcc through the resulting orbital motion.

Advantageously, the shear member may be moved closer to or further away from the rotating surface so as to allow the applied shearing force to be varied.

Alternatively, the rotating surface is not generally planar but is formed as an interior surface of a cone, dome or canopy shaped support element. Preferably, the support element has a substantially circular cross-section when sectioned along a plane which is substantially perpendicular to the axis of rotation. Reactant is applied by way of the feed means to a central inner portion of the surface before being spread across the surface in the form of a thin film. In this embodiment, the shear member may take the form of a plug shaped such that, when the plug is mounted coaxially at least partially within the support element, a circumferential region of the plug applies a shearing force to the reactant or product film on the rotating interior surface while still allowing the support element to rotate freely. In other words, the plug is preferably shaped so that its circumferential region matches the shape of the interior rotating surface but is slightly smaller so as to allow free rotation of the support element. In some embodiments, the plug may be generally disc shaped, having a circumferential wall shaped so as preferably to complement the shape of the interior rotating surface along the axis of rotation. Alternatively, the plug may also be cone, dome or canopy shaped, and sized so that it may be coaxially mounted at least

partially inside the support element and such that an external surface of the plug complements the interior rotating surface of the support element. In all of these embodiments, the plug is preferably movable along the axis of rotation of the support element so as to adjust its proximity to the interior rotating surface and thus to adjust the applied shearing force. The plug may be held stationary with respect to the support element, or may be rotated in the same or in an opposite direction, provided that there is relative movement between the plug and the interior rotating surface.

By applying a shearing force in this way, excellent mixing of reactants may be achieved. The reactor of the present invention is particularly suited to forming emulsions of two or more generally immiscible fluids and to promoting intimate mixing of all types of fluids.

The shear member may be coated or otherwise provided with a catalyst, typically a heterogeneous catalyst, as may the surface of the support element. Examples of suitable catalytic coatings include nickel, palladium, platinum and various alloys. The application of a catalyst can help to promote chemical reactions in the reactor.

The part of the shear member which contacts the film of reactant on the rotating surface may be generally smooth, or may be provided with discontinuities such as a mesh, grid or corrugations which serve to enhance surface areas.

The shear member may also be provided with heating or cooling means, such as an electric heater or a heat exchanger thermally connected thereto.

The axis of rotation of the rotating surface or support element may be substantially vertical, in which case gravity tends to pull reactants downwardly with respect to the surface or support member. This may be advantageous with less viscous reactants. Alternatively, the axis of rotation may be generally horizontal, which can achieve improved mixing of reactants provided that these are appropriately retained on the first surface of the support member.

Any suitable feed means may be provided to feed the at least one reactant onto the rotating surface. For example, the feed means may comprise a feed distributor in the form of a "shower head", a "necklace" of outlets or a simple, preferably adjustable, single point introduction such as a "hose-pipe type" feed means. Preferably, the feed means comprises a feed distributor having a plurality of uniformly spaced outlets for

the at least one reactant on to the rotating surface as hereinbefore defined. The feed means may also include means for applying UV, IR, X-ray, RF, microwave or other types of electromagnetic radiation or energy, including magnetic and electric fields, to the reactants as they are fed to the trough, or may include means for applying vibration, such as ultrasonic vibration, or heat.

The feed means may be provided at any suitable position with respect to the rotating surface which allows feed of the reactant. For example, the feed means may be axially aligned with the rotating surface for axial feed. Alternatively, the feed means may be positioned such that the feed is spaced from the axis of the rotating surface. Such a position may lead to more turbulence and an enhanced mixing effect.

Advantageously, the rotating surface includes a trough into which the at least one reactant is supplied by the feed means.

The depth of the trough in the rotating surface may be selected in accordance with reaction requirements. For example, for photochemical reactions in which UV light is shone onto the reactant, it is preferred for the trough to be relatively shallow, for example having a depth of the same order of magnitude or within one order of magnitude as the expected thickness of a film of reactant formed across the first surface of the support element when rotating at an appropriate speed.

In one embodiment, feed means may comprise a single feed to each trough which is preferably situated on or co-axial with the axis of rotation of the rotating surface. In this embodiment, reactant flows from the feed outlet into the trough and is subsequently spread out of the trough on to the rotating surface by centrifugal force. In a preferred embodiment, the rotating element as hereinbefore defined comprises a trough situated on the axis of rotation.

The trough as hereinbefore defined may be of any suitable shape such as continuous or annular. For example it may have a continuous concave surface comprising part of a sphere, such as a hemispherical surface, or it may have an inner surface joined to the rotating surface by at least one connection wall or at least two, in the case where the trough is annular. The inner surface and connection wall may be of any form which allows the function of a trough to be fulfilled. For example the inner surface may be parallel to the rotating surface or concave or convex. The connection wall may comprise a single circular or ovoid wall or a plurality of straight walls. The

walls may diverge or converge towards the rotating surface.

Preferably, a single circular wall is provided which converges towards the rotating surface to form an undercut trough. This shape generates a reservoir which enhances a circumferential distribution of the reactant or heat transfer fluid flow. Alternative means for forming an undercut trough are also envisaged. For example, where the trough is generally annular in shape, an outer wall may be provided as above, and an inner wall having any suitable shape may serve to define an inner edge to the trough. The undercut portion of the trough should generally be provided as an outer wall so as to help prevent uncontrolled egress of reactant or heat transfer fluid from the trough to the first or second surface under the influence of centrifugal force as the support element is rotated.

Advantageously, a matrix may be provided in the trough so as to help reactant or heat transfer fluid present in the trough to rotate with the support element, thereby helping to achieve substantially uniform flow from the trough across the first or second surface. The matrix may be in the form of a plug of fibrous mesh, such as metal or plastics wool, or may take the form of a plurality of projections which are secured to an inner surface of the trough. Other matrix means will be apparent to the skilled reader. In some embodiments, the matrix is manufactured of a material which is inert with respect to the at least one reactant or the product and which is not significantly affected by temperature and other variable process conditions. Alternatively, the matrix may be made of a material which does interact with the at least one reactant or the product, such as a heterogeneous catalyst (e.g. nickel, palladium or platinum or any suitable metal or alloy or compound thereof). Where the matrix is made out of an electrically conductive material, it may be possible to supply an electric current therethrough and thus to provide heating means for heating the at least one reactant within the trough.

In a further embodiment, there may be provided a plurality of feeds adapted selectively to supply one or more reactants to a plurality of troughs formed in the first surface. For example, where the support element is generally disc-like and has a substantially central axis of rotation, there may be provided a first central trough centred on the axis of rotation and feed means for supplying at least one reactant to the first trough, and at least one further trough, preferably also centred on the axis of rotation and having an annular configuration, the at least one further trough being provided with feed means for supplying a second reactant, which may be the same as

or different from the first reactant, to the at least one further trough. It will be apparent to the skilled reader that a plurality of troughs may be provided in a similar manner on support elements with shapes other than generally disc-likc.

By providing a plurality of troughs and feeds, a sequence of reactions can be performed across the first surface of the support element. For example, two reactants may be supplied to the first trough in which some mixing and reaction will take place. As the support element rotates, the reactants will spread from the first trough to the first surface of the support element, where further reaction and mixing takes placc, and thence into a second annular trough concentric with the first trough. A third reactant may then be supplied to the second trough, and further mixing and reaction will take place as the third reactant and the two initial reactants and any associated product are spread from the second trough onto the first surface of the support element for further mixing and reaction. Because the direction of travel of the reactants and products is outwards from the axis of rotation, a controlled series of reactions can be carried out across the first surface of the support member.

Any suitable collection means may be provided for collection of the product as it leaves the rotating surface at its periphery. For example, there may be provided a receptacle in the form of a bowl or trough at least partially surrounding the rotating element or other fixed part of the apparatus. The collection means may additionally comprise a deflector positioned around the periphery of the rotating surface to deflect product into the collection means. The deflector is preferably positioned at an acute angle to the rotating surface.

The components of the collection means, such as the bowl or trough or deflector, may be coated or otherwise provided with a heterogeneous catalyst appropriate to the reactants being reacted on the support element, or may even consist entirely of a material which acts as a heterogeneous catalyst. Furthermore, the components of the collection means may be heated or cooled to a predetermined temperature so as to enable control over reaction parameters, for example by serving to halt the reaction between reactants as these leave the first surface in the form of product. Feed means for supplying a reactant to the product leaving the first surface may also be provided. For example, there may be provided feed means for feeding a quenching medium to product in the collection means so as to halt chemical or other reactions between reactants when these have left the first surface.

The collection means may further comprise outlet means of any suitable form. For example, there may be a single collection trough running around the periphery of the disc or a collection bowl partially surrounding the rotating element.

Outlet means may also be provided in the collection means and these may take the form of apertures of any size and form situated at any suitable position of the collection means to allow egress of the product. In one preferred embodiment, the outlet means are situated to allow vertical egress of the substrate in use.

Alternatively, the collection means may comprise an outer wall provided at the periphery of the support element so as to prevent product from being thrown from the first surface, and at least one pitot tube which extends into the product which is restrained at the periphery of the support element by the outer wall. The outer wall may converge generally towards the axis of rotation of the support member so as better to retain product while the support element is undergoing rotation, although other wall configurations, such as generally parallel to or divergent from the axis of rotation may also be useful.

Embodiments of the present invention may include multiple support elements, which may share a common axis of rotation and which may be mounted on a single rotatable shaft, or which may be provided with individual rotatable shafts. The collection means associated with any given support element may be connected to the feed means associated with any other given support element so as to link a number of support elements in series or parallel. In this way, a reaction may be conducted across a number of support elements in series or parallel. The collection means of a first support member may be directly connected to the feed means of a second support member, or may be connected by way of a processing unit such as a pump, extruder, heater or heat exchanger or any other appropriate device. This is especially useful when dealing with viscous products, such as those which are obtained in polymerisation reactions, since the viscous product of a first support element may be processed so as to acquire more favourable physical characteristics before being used as the reactant feed for a second support element.

For example, where the collection means comprises an outer wall on the first surface of the support element as described above, a number of support elements may be coaxially mounted on a single rotatable shaft so as to form a stack of support elements. A reactant feed is led to the trough of a first support element, and a

collector in the form of a pitot tube has its tip located near the first surface of the first support element in the vicinity of the wall so as to take up product from this region. An end of the pitot tube remote from the tip is led to the trough of a second support element so as to allow the product of the first support element to serve as the reactant for the second support element, thereby allowing a number of reactions to take place in series. Alternatively, a number of parallel feeds may supply the same at least one reactant simultaneously to the troughs of a number of support elements and a number of parallel pitot tube collectors may gather product from a peripheral region of each support element, thereby allowing a reaction to take place across a number of support elements in parallel.

It is also envisaged that product collected from the periphery of a support element may be recycled as feed for that support element. This is useful for processes requiring an extended contact time for the reactants. The product may be fully or only partially recycled, depending on requirements.

Reference herein to a rotating surface is to any continuous or discrete planar or three dimensional surface or assembly which rotates approximately or truly about an axis, and preferably is reference to an approximate or true rotating surface of revolution. An approximate rotating surface of revolution may comprise an asymmetric axis and/or deviation in the surface body and/or circumference creating an axially or radially undulating surface of revolution. A discrete surface may be in the form of a mesh, grid, corrugated surface and the like.

Reference herein to a substantially radially outward flowing film as hereinbefore defined is to any fluid film which may be created by dynamic contact of the fluid phase reactant and the rotating surface as hereinbefore defined, suitably the fluid phase reactant is contacted with the rotating surface at any one or more surface locations and caused to flow outwardly by the action of centrifugal force. A film may be a continuous annulus or may be a non-continuous arc at any radial location. The substrate may provide a plurality of films in dynamic contact with a rotating surface as hereinbefore defined.

For example processes requiring extended contact time may be carried out in continuous manner with use of a recycle of fluid exiting at the periphery of the rotating surface towards the axis of the rotating surface enabling sequential passes of fluid across the surface. In continuous steady state operation an amount of fluid

exiting the surface may be drawn off as product and an amount may be returned by recycle for further conversion with an amount of fresh reactant feed.

The process of the invention as hereinbefore defined may operated in a single or plural stages. A plural stage process may comprise a first pre-process stage with further post-process or upgrading stages, and may be carried out batchwise with use of a single rotating surface as hereinbefore defined or may be carried out in continuous manner with multiple rotating surfaces in series.

Second or more reactants may be added to the feed reactant as it passes from one rotating assembly to the next or be added directly to the rotating assembly anywhere between the axis of rotation or the exit from the assembly. In certain cases a multi-step process may be achieved by reactant addition or additions between the axis of rotation and the exit of a single rotating assembly to achieve more than one process step in a single pass. It is also possible to have different regions of the rotating surface at different temperatures and conditions and have different surface geometries as appropriate to the process needs.

It will be apparent that the process of the invention may be controlled both by selection of a specific rotating surface for the support element and by selecting process variables such as temperature, speed of rotation, rate of reactant feed, conversion time and the like. Accordingly the process of the invention provides enhanced flexibility in process control including both conventional control by means of operating conditions, and additionally control by means of rotating surface type.

The apparatus may further comprise any suitable control system. Such a control system may regulate the temperature or contact time of reactants by means of speed of rotation, rate of substrate feed and other process parameters to obtain an optimum result.

The apparatus as hereinbefore defined may comprise means for optimising process conditions. For example, means for imparting an additional movement to the rotating surface, and thus to the reactant, may be provided. Such movement could be in any desired plane or plurality of planes and preferably comprises vibration. Any suitable vibration means may be provided, such as flexible mounting of the surface or off centre mounting, both inducing passive vibration or active vibration means, such as a mechanical element in contact with the rotating element and vibrating in a

direction parallel to the rotating element axis. Preferably a passive vibration means is provided in the form of off centre mounting of the rotating element on its axis of rotation. Vibration may alternatively be provided by an ultrasonic emitter in contact with the rotating element for vibration in any desired plane or plurality of planes.

The rotating surface may have any shape and surface formation to optimise process conditions. For example the rotating surface may be generally planar or curved, frilled, corrugated or bent. The rotating surface may form a cone or be of generally frustoconical shape.

In one preferred embodiment the rotating surface is generally planar and preferably generally circular. The periphery of the rotating surface may form an oval, rectangle or other shape.

In another preferred embodiment the rotating surface is provided as the inner surface of a cone. The apparatus may comprise at least one cone and at least one other rotating surface or at least one pair of facing cones positioned so as to allow a two stage process with one or more reactants fed to each cone. Preferably product exits a smaller cone (or other surface of rotation) in a spray on to the surface of a larger cone (or other surface of rotation) by which it is at least partially surrounded and for the surface of which a further reactant is fed by feed means as hereinbefore defined, to allow mix of the product and reactant on the larger rotating surface. Preferably, means are provided such that the two cones counter rotate. Such an arrangement enhances mixing and intimate contact of the reactants and reduces the required physical contact time. Alternatively, means are provided such that the cones co-rotate or one is stationary.

A rotating surface of any shape and surface formation as hereinbefore defined may be provided with surface features which serve to promote the desired process. For example, the surface may be micro or macro profiled, micro or macro porous, non stick, for example may have a release coating, may be continuous or discontinuous and may comprise elements such as mesh, for example woven mesh, reticulate foam, pellets, cloth, pins or wires, for enhanced surface area, enhanced or reduced friction effect, enhanced or reduced laminar flow, shear mixing of recirculation flow in axial direction and the like.

In one preferred embodiment, mixing characteristics of the rotating surface are

enhanced by the above features or the like provided on or in the rotating surface. These may be provided in any suitable regular or random arrangement of grids, concentric rings, spider web or like patterns which may be suitable for a given application.

Alternatively or additionally to any other surface feature, radially spaced pins in the form of circles or segments of circles may be provided.

In another preferred embodiment, a porous surface coating is provided, which aids processing of certain reactants. Such a coating may be provided in combination with any other of the aforementioned surface features.

Surface features in the form of grooves may be concentric or may be of any desired radially spaced form. For example, the grooves may form "wavy" or distorted circles for maximised mixing.

Grooves may be parallel sided, or may have one or both sides which diverge to form undercut grooves or which converge to form tapered grooves. Preferably, the grooves are undercut to promote mixing.

Grooves may be angled to project towards or away from the axis of the rotating surface to enhance or reduce undercut or taper.

Energy transfer means may be provided for the rotating surface or reactant or product as hereinbefore described. For example heating means may be provided to heat the reactant, for example, as part of the feed means. Additionally, or alternatively heating means may be provided to heat the rotating element in the form of radiant or other heaters positioned on the face of the rotating element which does not comprise the rotating surface for conversion. Preferably, radially spaced, generally circular radiant heaters are provided.

Any preferred cooling or quenching means may be provided in a suitable position to cool the reacted substrate. For example cooling coils or a heat sink may provide cooling by heat exchange, or a reservoir of quench may provide cooling or reaction termination by intimate mixing in the collection means.

For a better understanding of the present invention and to show how it may be carried

into effect, reference shall now be made by way of example to the accompanying drawings, in which:

FIGURE 1 shows a spinning disc apparatus in schematic form;

FIGURE 2 shows a spinning disc provided with a dome shaped shear member;

FIGURE 3 shows a spinning cone with a plug shear member;

FIGURE 4 shows a detail of a spinning disc having a central trough; and

FIGURE 5 shows a detail of a spinning disc having an annular trough.

Figure 1 illustrates a spinning disc apparatus of the present invention. The apparatus is enclosed in vessel (1) having at its axis a drive shaft (2) supporting a spinning disc (3). Feed means (4) provides reactant to the surface (5) of the disc (3) about its axis (6). Rotation of the disc (3) causes reactant to flow radially outwards, whereby it contacts the surface (5) of the spinning disc (3) and forms a film (17) thereacross. A stationary, annular shear member (18) is located just above the surface (5) such that a basal portion (19) of the shear member (18) contacts the film (17) and applies a shearing force thereto when the disc (3) is rotated. Fluid is collected at the peripheral edges of the disc (3) by means of collection trough (7) and may be rapidly quenched by means of cooling coils (8). A skirt (9) prevents meniscal draw back of fluid contaminating the drive shaft mechanism. Inlet means (10) enable controlled environment conditions to the provided, for example a nitrogen atmosphere. Outlet vent means (11) enable the venting of atmospheric gases or gases evolved during operation. Observation means are provided by means of windows (12) to observe the progress of the conversion.

The apparatus of Figure 1 may be started up and operated as described in Example 1 below. In the case that the process is an exothermic conversion, cooling coils (8) may be used to quench the collected product in the trough (7). The spinning disc (3) is provided with heating coils (not shown) which may be used to initiate or maintain conversion. The disc (3) or the reactor vessel (1) may be provided with a source of radiation, means for applying an electric or magnetic field and the like as described, at or above the disc surface (5) or at the wall of the reactor vessel (1).

Figure 2 shows a spinning disc (3) having a surface (5) and mounted on a drive shaft (2) which defines an axis of rotation (6). A feed pipe (4) supplies reactant (15) to a central trough (14) (see below) from where the reactant (15) spills onto the surface (5) in the form of a film (17). A shear member in the form of a canopy (20) having a peripheral basal surface (21) is mounted over the disc (3) such that, in operation, the basal surface (21) contacts the film (17) and applies a shearing force thereto by virtue of relative movement between the basal surface (21) and the rotating surface (5). This shearing force helps to promote intimate mixing of the reactant (15). The canopy (20) may be fixed relative to the disc (3), or may be adapted to rotate in either the same or the opposite direction, provided always that there is relative movement between the basal surface (21) and the rotating surface (5). Furthermore, the spacing between the basal surface (21) and the rotating surface (5) may be adjusted by moving the canopy (20) up or down so as to vary the shearing force applied to the film (17). Product (27) is thrown from the rotating surface (5) at a peripheral portion thereof for collection.

Figure 3 shows a beaker shaped support element (22) having an internal surface (23) with divergent sides and mounted on a horizontal drive shaft (24). Reactant (15) is supplied to a trough (14) in a base region of the support element (22) from where it spills out onto the surface (23) in the form of a film (17) when the support element (22) is rotated. A disc shaped plug (25) having a peripheral surface (26) complementary to the surface (23) is mounted in the support element (22) so as to contact the film (17) when the support element (22) is rotated, thus applying a shearing force. The plug (25) may be stationary or may rotate with or against the surface (23), provided always that there is relative movement between the two. By moving the plug back and forth along the axis of the drive shaft (24), the spacing between the peripheral surface (26) and the internal surface (23) may be varied so as to vary the applied shearing force. Product (27) is thrown from the inner surface (23) at a mouth portion of the support element (22) for collection.

In Figure 4 there is shown an axially located central trough (14) which is continuous and forms a well situated on the axis of rotation (6) of the rotating surface (5) of a disc (3). Rotation causes reactant or heat transfer fluid (15) supplied by the feed means (4) to flow to the wall and form an annular film (16) within the trough (14). The annular film (16) then spills over onto the surface (5) of the disc (3) to form a film (17) on the surface (5).

In Figure 5 the trough (13) is annular and forms a channel co-axial about the axis of rotation (6) of the disc (3). Rotation assisted by the trough profile causes reactant or heat transfer fluid (15) to flow into the trough (13) and to the wall thereof and form an annular film (16) within the trough (13) before spilling over onto the surface (5) of the disc (3) in the form of a film (17).

Example 1 – Polymerisation of ethylene using a catalyst coated disc

Phillips catalyst was coated onto the surface of a spinning disc apparatus using methods as described hereinbefore. The coated disc was mounted in a spinning disc apparatus.

The spinning disc apparatus used is shown in diagrammatic form in Figure 1. The main components of interest being:

i) Top Disc – A smooth brass disc of thickness 17mm and diameter 500mm capable of rotating around a vertical axis.

ii) Liquid Distributor – A circular copper pipe of diameter 100mm, positioned concentrically over the disc, sprayed fluid vertically onto the disc surface from 50 uniformly spaced holes in the underside. Flowrate was controlled manually by a valve and monitored using a metric 18 size, stainless steel float rotameter. A typical fluid flow rate was 31.3cc/s.

iii) Motor – A variable speed d.c. motor capable of rotating at 3000 rpm was used. The rotational speed was varied using a digital controller calibrated for disc speeds between 0 and 1000 rpm. A typical rotational speed was 50rpm.

iv) Radiant Heaters – 3 radiant heaters (each consisting of two elements) spaced equally below the disc provided heat to the disc. The temperature was varied using a temperature controller for each heater. Each heater temperature could be controlled up to 400°C. Triac regulators were used to control the speed of the controller response. (These remained on setting 10 throughout the tests).

v) Thermocouples and Datascanner – 16 K-type thermocouples embedded in the top disc gave an indication of the surface temperature profile along the disc radius. Odd numbered thermocouples 1 to 15 inclusive were embedded from underneath the disc to a distance 3mm from the upper disc surface. Even numbered thermocouples,

2 to 16 inclusive were embedded from underneath the disc to a distance 10mm from the upper disc surface. Each pair of thermocouple, i.e. 1 & 2 and 3 & 4 and 5 & 6 etc., were embedded adjacently at radial distances of 85mm, 95mm, 110mm, 128mm, 150mm, 175mm, 205mm and 245mm respectively (see Figure 3). The thermocouples were connected to the datascanner which transmitted and logged the data to the PC at set intervals using the DALITE Configuration and Monitoring Software Package.

vi) Manual Thermocouple – A hand-held K-type thermocouple was used to measure the bulk fluid temperature on top of the disc.

The rig was used in two arrangements. In one arrangement, feed was constantly added and the heated product was sent to the collection trough. In an alternative arrangement the rig was assembled with a recycle.

The spinning disc apparatus of Figure 1 was started up and temperature and rotational speed adjusted. When steady stage was achieved gaseous ethylene was fed to the revolving catalyst coated disc surface at it axis. Product was withdrawn in the collection trough at the periphery of the disc. Analysis revealed the product was high grade polyethylene.

Further advantages of the invention are apparent from the foregoing.

Claims

CLAIMS:

1. A reactor apparatus including a support element adapted to be rotatable about an axis, the support element having a surface and feed means associated therewith for supplying at least one reactant to the surface, characterised in that there is further provided a shear member which is disposed in close proximity to the surface so as, in use, to impart a shearing force to the reactant on the surface when the support element is rotated.

2. A reactor as claimed in claim 1, wherein the surface is substantially planar.

3. A reactor as claimed in claim 1 or 2, wherein the surface is substantially circular.

4. A reactor as claimed in any preceding claim, wherein the shear member comprises at least one generally elongate member which, in use, contacts the reactant on the surface of the support element.

5. A reactor as claimed in any one of claims 1 to 3, wherein the shear member comprises a generally planar surface which, in use, contacts the reactant on the surface of the support element.

6. A reactor as claimed in any one of claims 1 to 3, wherein the shear member comprises a dome or canopy having a peripheral basal surface which, in use, contacts the reactant on the surface of the support element.

7. A reactor as claimed in any one of claims 1 to 3, wherein the shear member comprises a cylindrical or tubular member having a peripheral basal surface which, in use, contacts the reactant on the surface of the support element.

8. A reactor as claimed in claim 1, wherein the surface is formed as an interior surface of a cone, dome or canopy shaped support element.

9. A reactor as claimed in claim 8, wherein the interior surface of the support element has a substantially circular cross-section when sectioned along a plane substantially perpendicular to the axis of rotation.

10. A reactor as claimed in claim 8 or 9, wherein the shear member comprises a plug having a circumferential surface shaped such that, when the plug is mounted coaxially and at least partially within the support element, the circumferential surface contacts the reactant on the interior surface of the support element when the reactor is in use while allowing the support element to rotate freely.

11. A reactor as claimed in claim 10, wherein the plug is generally disc shaped.

12. A reactor as claimed in claim 10, wherein the plug is cone, dome or canopy shaped.

13. A reactor as claimed in any preceding claim, wherein the shear member is adjustable so as to vary its spacing from the surface of the support element.

14. A reactor as claimed in any preceding claim, wherein the shear member is held stationary during rotation of the support element.

15. A reactor as claimed in any one of claims 1 to 13, wherein the shear member is adapted, in use, to rotate in an opposite direction to the support element.

16. A reactor as claimed in any one of claims 1 to 13, wherein the shear member is adapted, in use, to rotate in the same direction as the support element but at a different rotational speed.

17. A reactor as claimed in claim 15 or 16 depending from any one of claims 1 to 7, wherein the shear member has an axis of rotation which is offset from the axis of rotation of the support element.

18. A reactor as claimed in any preceding claim, wherein the shear member is coated or otherwise provided with a heterogeneous catalyst.

19. A reactor as claimed in any preceding claim, wherein a surface of the shear member which, in use, contacts the reactant, is substantially smooth.

20. A reactor as claimed in any preceding claim, wherein a surface of the shear member which, in use, contacts the reactant, is provided with discontinuities which enhance its surface area.

21. A reactor as claimed in claim 20, wherein the discontinuities comprise a mesh, grid or corrugations.

22. A reactor as claimed in any preceding claim, wherein the shear member is provided with temperature control means.

23. A reactor as claimed in claim 22, wherein the temperature control means is a heater or heat exchanger.

24. A reactor as claimed in any preceding claim, wherein the axis is substantially parallel to a direction of action of terrestrial gravity.

25. A reactor as claimed in any one of claims 1 to 23, wherein the axis is inclined with respect to a direction of action of terrestrial gravity.

26. A reactor as claimed in any one of claims 1 to 23, wherein the axis is substantially perpendicular to a direction of action of terrestrial gravity.

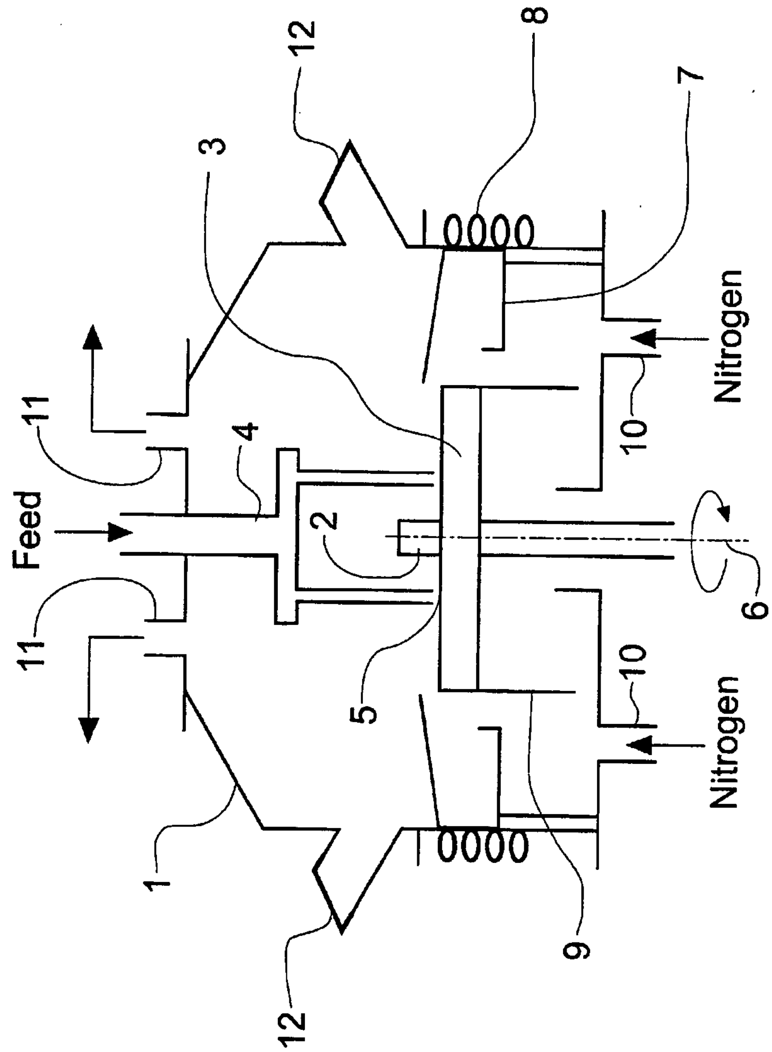

Fig. 1

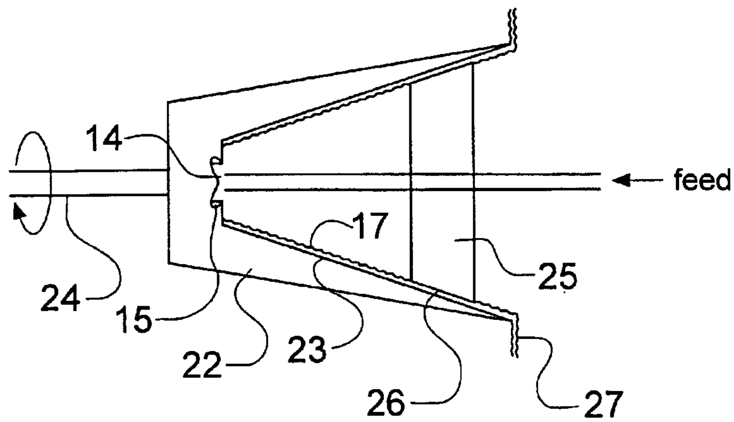

Fig. 3

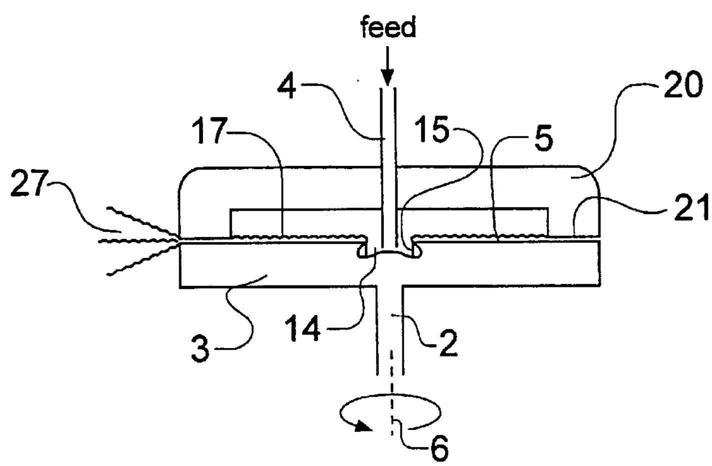

Fig. 2

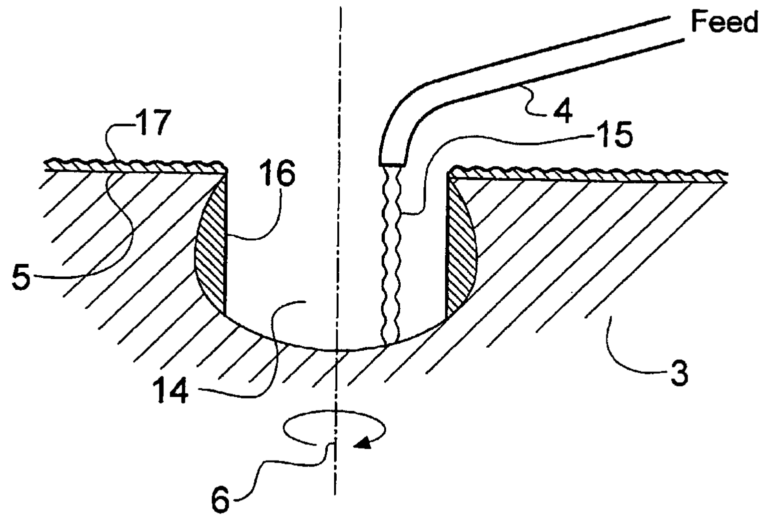

Fig. 4

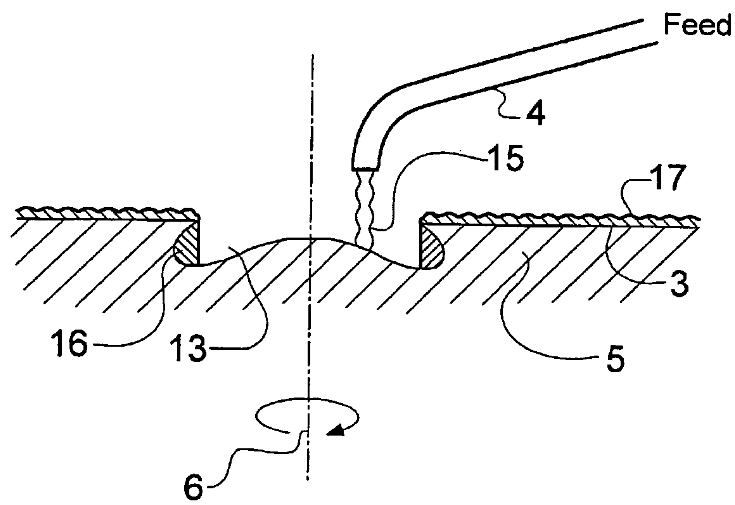

Fig. 5

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UNIVERSITY OF NEWCASTLE [/]; (). RAMSHAW, Colin [/]; (). JACHUCK, Roshan, Jeet, Jee [/]; (). RAMSHAW, Colin [/]; (). JACHUCK, Roshan, Jeet, Jee [/]; (). HARRISON GODDARD FOOTE ; ().

(54) Title: ROTATING SURFACE OF REVOLUTION REACTOR WITH SHEARING MECHANISMS
(54) Titre: SURFACE TOURNANTE DE REACTEUR ROTATIF COMPORTANT DES DISPOSITIFS DE CISAILLEMENT

(57) Abstract

A reactor including a rotatable disc (3) having a surface (5) onto which reactant (15) is supplied by way of a feed (4). The disc (3) is rotated at high speed, and the reactant (15) spills over the surface (5) so as to form a film (17). A shear member (18, 20) is mounted close to the surface (5) so as to contact the film (17) during operation of the reactor, thereby applying a shearing force to the reactant (15) so as to aid mixing.

(57) Abrégé

L'invention concerne un réacteur comportant un disque tournant (3) qui présente une surface (5) sur laquelle un réactif est appliqué au moyen d'un dispositif d'apport (4). On fait tourner le disque (3) à grande vitesse, et le réactif (15) se répand sur la surface (5) de manière à former un film (17). Un élément de cisaillement (18, 20) monté à proximité de la surface (5) de façon à entrer en contact avec le film (17) au cours de l'exploitation du réacteur, permet d'appliquer une force de cisaillement au réactif (15) pour faciliter le mélange.

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(71) Applicant (*for all designated States except US*): **UNIVERSITY OF NEWCASTLE** [GB/GB]; 6 Kensington Terrace, Newcastle upon Tyne NE1 7RU (GB).

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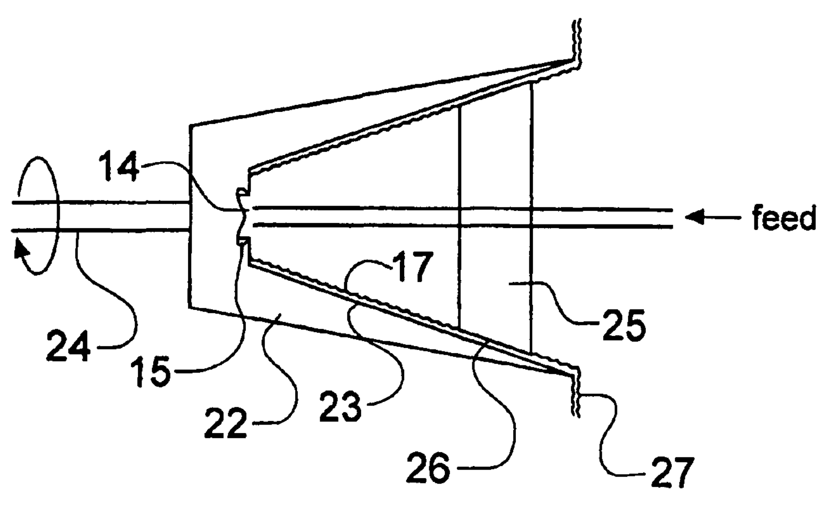
[Continued on next page]

(54) Title: ROTATING SURFACE OF REVOLUTION REACTOR WITH SHEARING MECHANISMS



(57) Abstract: A reactor including a rotatable disc (3) having a surface (5) onto which reactant (15) is supplied by way of a feed (4). The disc (3) is rotated at high speed, and the reactant (15) spills over the surface (5) so as to form a film (17). A shear member (18, 20) is mounted close to the surface (5) so as to contact the film (17) during operation of the reactor, thereby applying a shearing force to the reactant (15) so as to aid mixing.

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INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B01J19/18 B01J19/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B01J B01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 624 999 A (LOMBARDI ALESSANDRO, BARINI GERALDO, D'ANTONIO CARMINE, GUSI STEFANO) 29 April 1997 (1997-04-29) column 14, line 20 -column 18, line 25 column 25, line 62 -column 27, line 2 figures 3-6	1-7, 14-16, 19,20, 24,26
X	US 3 831 907 A (CLAES FRANS HENRI) 27 August 1974 (1974-08-27) column 4, line 36 -column 5, line 10 column 6, line 29 - line 36 column 7, line 7 - line 19 column 7, line 49 -column 8, line 27 figures 1,4	1,8,9, 14,15, 19-26

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
3 August 2000	11/08/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Vlassis, M

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INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 00/00523

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 511 414 A (MATSUI FUMIO, AONO SHIN, SAKAI HIROSHI, HATTORI KATSUHIDE, KAKINO SHIGERU) 16 April 1985 (1985-04-16) column 4, line 26 - line 61 claim 1; figure 3 ---	1-3,24
A	GB 2 108 407 A (LEUNA WERKE VEB) 18 May 1983 (1983-05-18) the whole document ----	1-3,14, 22-24

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INTERNATIONAL SEARCH REPORT

Information on patent family members

Inte. onal Application No
PCT/GB 00/00523

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

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International application No. PCT/GB00/00523	International filing date *(day/month/year)* 17/02/2000	Priority date *(day/month/year)* 17/02/1999
International Patent Classification (IPC) or national classification and IPC B01J19/18		
Applicant NEWCASTLE UNIVERSITY VENTURES LIMITED et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.

2. This REPORT consists of a total of 4 sheets, including this cover sheet.

   ☒ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

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3. This report contains indications relating to the following items:

   I ☒ Basis of the report
   II ☐ Priority
   III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
   IV ☐ Lack of unity of invention
   V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations suporting such statement
   VI ☐ Certain documents cited
   VII ☐ Certain defects in the international application
   VIII ☐ Certain observations on the international application

Date of submission of the demand	Date of completion of this report
15/09/2000	08.05.2001
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